Ecofriendly pretreatment of cotton knit fabrics by ultrasonic irradiation and reusing bath chemicals

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Abstract

Bangladesh's cotton knit industries contribute significantly to the country's economy, accounting for more than 40% of total annual export. Efficient water use in knit dyeing might result in considerable water savings and environmental sustainability. Textile wet processing consumes a lot of energy (water, gas, and electricity), generates a lot of waste, and has significant chemical costs. The cotton knit fabric used in this study was pretreated and dyed utilizing ultrasonication at a lower temperature than conventional pretreatment and dyeing techniques in an attempt to establish ecofriendly wet processing in the textile industry. For pretreatment and dyeing, bath chemicals were reused 1–3 times, and several fabric attributes including whiteness index, weight loss (%), bursting strength, color fastness to light, washing, perspiration, and rubbing were tested and compared to the traditional method. By dying with reactive dyes, the feasibility of color matching using ultrasonic aided pretreated fabric with and without reusing bath chemicals was determined. The whiteness index of the low temperature sonicated scoured and bleached fabric was found to be acceptable, with relatively low weight loss; nevertheless, the bursting strength was found to be enhanced. Color fastness to light, washing, perspiration, and rubbing of ultrasonicated dyed knitted fabric at lower temperatures than conventional techniques were found to be comparable, showing that there was no color deterioration during ultrasonication. FT-IR spectroscopy and scanning electron microscopy (SEM) revealed no significant changes in the chemical composition of cellulose or the fabric shape of pretreated and dyed cotton knit fabric after ultrasonication.

1. Introduction

In its numerous processing steps, the textile knit industry uses a substantial quantity of water and chemicals, resulting in a significant amount of effluent released into the environment (Kaneda & Bietsch 2015). Textile wet processing is ranked second among freshwater consumers and polluters because it has a significant environmental effect, making it one of the most environmentally harmful sectors (Ibrahim et al. 2019). To ensure the successful implementation and acceptance of cleaner production techniques and practices in the textile industry, production, user, and disposal ecologies should be considered (Hannan et al. 2018a, 2019). Combining two or more wet processes in a single step, for example, and reusing chemicals using alternative ways can save water, energy, and chemical usage, as well as reduce effluent load and productivity (Bhuiyan et al. 2016, Eid & Ibrahim 2021).

Scouring/bleaching helps remove natural and non-fibrous impurities from fabric surfaces while also destroying natural color, eliminating uneven coloring (Karmakar 1999). Industrially, these pollutants are removed in the same bath, and textiles are treated at high temperatures with NaOH and peroxide. Scouring/bleaching is an environmentally burdensome process since it requires a large amount of alkali and an extensive rinse operation that pollutes the washing effluent with environmentally hazardous chemicals (El Shafie et al. 2009). Completion of an uncontaminated dying technique is one of the requirements for successfully trading textile products (Tunç et al. 2009, Zhang & Zhang 2015). Knit batch dyeing techniques, which may be done in three steps, take into account dosing time, temperature, pH, pressure, and cycle time. (a) dye depletion in the presence of dye adsorption, (b) alkali-induced fixxing, and (c) unfix dye wash-off from surface material (Chakraborty & Ahmad 2022).

Conventional fabric dyeing consumes high amount of water and required a lot of energy (Wijesena et al. 2015). There has been a growing interest in employing ultrasonic irradiation to produce mechanical energy in textile wet processing since the early 1950s. Compressions and rarefactions caused by ultrasonic wave propagation (known as cavitation) are commonly attributed to the majority of physical and chemical processes that accelerate chemical propagation and reactions that require less power to initiate, making it an energy and time-saving technique for sustainable production (Abou-Okeil et al. 2010). Ultrasound energy, as a renewable energy source, has the potential to lower the global cotton-textile industry’s water impact and help the UN reach its Sustainable Development Goal. Ultrasound can be used to replace expensive heat energy and chemicals in textile wet processing (Gallego-Juarez 2010). During ultrasonication cavitation, bubbles form, acting as a micro-reactor for effective dye uptake (Perincek et al. 2009). When an ultrasonic wave passes through a dye solution, cavitation forms almost instantly (Nagaje & Hulle 2015). As a result of the ultrasonic treatment, the dyeing rate and homogeneous color absorption on the fabric are increased (Carr et al. 1996). The use of ultrasound during the pretreatment and dying process on cotton knitted fabric improves dye uptake percentages (Harifi & Montazer 2015). De-aggregation of dye molecules leads to greater dye diffusion and maybe aids dye fiber link fixxing, according to other observations.
It is typical practice to reuse some type of textile wastewater before sending it to a ETP to treat and recycle (Abu Shaid et al. 2013). The effluent from four phases of the knit dyeing process was discovered to be suitable for use in the new scouring bath. The water from the last rinse in a batch dyeing procedure is particularly clean and may be utilized for washing or setting up the future dye baths straight away. With minimal initial expenses, this reuse method has the potential to save 10–30% of water (Siddique et al. 2017). In this work, we have investigated the influence of ultrasound on the pretreatment process and dyeing of 100% cotton knit fabric with reactive dye at lower temperature than conventional processes. The effect of the pretreatment was carefully evaluated by wettability test, percentage of weight loss calculation, and CIE whiteness measurements. We have also reused the pretreatment bath chemicals in sonication method for pretreatment and dyeing. Different fabric properties were compared with traditional method and ultrasound assisted method in an attempt to develop energy and chemical efficient pretreatment and dyeing of cotton knitted fabric.

2. Material And Methods

Throughout the study, a 100% greige cotton plain knitted cloth from Micro Fiber, Narayanganj, Bangladesh was employed. The yarn count was 28/s (carded), the machine gauge was 24, the stitch length was 2.75 mm, and the GSM (gram per square meter) was 160. The wetting agents, detergent, caustic soda flakes (98.5%), hydrogen peroxide (35%), peroxide stabilizer, Glauber’s salt, sodium carbonate 99.2%, and reactive dyes used in this study were analytical grade and was obtained from E.Merck (India). Reactive dyestuffs (Dyechu-Red-3R-XE, Dysin-Chem Ltd, China) was used as obtained.

2.1 Conventional pretreatment and dyeing

A combined scouring-bleaching was performed following the recepie as given in Table 1 (Korkmaz et al.). Dyebath was prepared by adding specific amount of dyestuffs along with other auxiliaries in material-liquor ratio (M:L = 1:10). Infrared lab dyeing machine was utilized for pretreatment and dyeing.
2.2. Ultrasonication irradiation

Sweeping digital control ultrasonic cleaning units of 40 kHz frequency (Haoshun Ultrasonic Instrument Co., Ltd, Shenzhen, China) was used for ultrasonic irradiation having adjustable temperature control between 20–100°C.

2.3. FT-IR measurement

The Fourier Transform Infrared Spectroscopy (FT-IR, Shimadzu Corp, Japan) was used to investigate the cotton knitted fabric in order to validate the presence of various functional groups and to offer information on the materials' chemical composition. FTIR spectra in the 4000–500 cm\(^{-1}\) range were obtained and analyzed.

2.4. Scanning electron microscopic (SEM) analysis

The Field Emission SEM (JOEL JSM-7600F, Japan) was used to observe the surface topography of conventionally and sonicated scoured-bleached pretreated samples, as well as dyed samples from both conditions.

2.5. Wettability test

The water contact angle of the fabric sample was measured using an automated optical contact angle meter (Data physics, OCA20, Germany). The wetting time was calculated according to AATCC-79-2010 by measuring the time it took a water drop falling from a specified height to lose its specular reflection. All fabric samples were adjusted in the standard condition for 24 hours prior to measurement. After four experiments, the average value was calculated. Water absorption in under 5 seconds indicates wax removal and consequently good scouring (Sennur 2010).

2.6. Vertical wicking test
Wicking is a term that relates to the liquid transport behavior of fiber assemblies and therefore is important in defining the comfort qualities of the fabric. Vertical wicking tests on knit fabric were done in accordance with AATCC Test Methods 197–2011. The fabric sample was hung from a platform, and its bottom was brought into contact with a liquid reservoir set on a base stand. Capillary forces are formed when the liquid makes contact with the fabric's bottom, allowing the liquid to ascend against gravity (Duru & Candan 2013). The rate and amount of liquid rise are then recorded and evaluated for characterization. After 30 minutes, the allowable assessment range is 6 inches.

2.7. Calculation of weight loss

Weight loss happens during the pretreatment process, which involves removing the cuticle layer, protein, and pectin material from the α-glucose. The average weight loss is between 4 and 8% of the total weight (Aly et al. 2010). The most common use of a weight loss test is to determine the scouring impact. Scoured-bleached cotton samples were taken after complete oven drying at 105°C and subsequently conditioning under typical atmospheric conditions (65 ± 2% RH and 20 ± 2°C) for 8 hours to determine percent weight loss percentage (Asaduzzaman et al. 2016). Weighing the treated and untreated fabrics yielded the percentage of weight decrease. The formula for calculating weight loss is as follows:

\[ \text{Wt\%} = \frac{W_1 - W_2}{W_1} \times 100 \]

W1 and W2, respectively, are the fabric weights before and after treatment.

2.8. CIE Whiteness measurement

For each bleached sample, the CIE Whiteness Index (WI) value was determined using AATCC Test Method 110–1995. A Datacolor Spectrophotometer SF 600X was used to assess whiteness, using illuminant D65 and 10° observer (Tutak et al. 2011). CIE denotes for Commission internationale de l’éclairage [French], International Commission on Illumination [English].

2.9. Fabric strength test

The bursting strength of a fabric is the amount of pressure necessary to burst the fabric surface. When a certain amount of pressure is given to the fabric, it begins to expand simultaneously in all available directions. The fabric breaks after exceeding the bursting strength pressure limit as the applied pressure gradually increases (Ünal et al. 2010). It is expressed in pounds per inch 2 (lbs.) or kilograms per centimeter (kg/cm).

2.10. Color fastness

Colorfastness, a key criterion for dyeing performance, is the capacity of a color to adhere to a material, ensuring color resistance to fading (Vigo 2013). Colorfastness to wash, perspiration, rubbing, and light were all evaluated in this study. The approaches for assessing color fastness in textile materials include standardizing the dyeing process and visually comparing treated and untreated fabrics against a gray scale comprised of pairs of color chips with increasing color difference magnitude (Rashid et al. 2012). Four categories of colorfastness were evaluated during the trial, including colorfastness to wash, perspiration, rubbing, and light. ISO 105-C06-C2S:2010 1A, ISO 105×12:2001, ISO 105 E04:2013, and ISO105-B02:2014 were used to determine these properties (Hurren 2008). Different factors such as brightness, red-greenness scale, yellow-blueness scale, saturation, and hue angle, denoted as L*, a*, b*, c*, H, are used to measure color difference (Robertson 1990). CMC DE must be between 0.75 and 1 in order to be commercially acceptable (McDonald 1988). The Color Measurement Committee of the Society of Dyers and Colourists of Great Britain is represented by CMC. To match the shade with the reference sample, sonication is used to create three alternative shades.

3. Results And Discussion

3.1. FT-IR analysis

FT-IR spectra of pretreated cotton fabric acquired using conventional and sonication methods are depicted in Fig. 1. Generally, hydrogen bonding occurred between 3200 and 3600 cm⁻¹, however one medium peak was developed at 3850 cm⁻¹ in this
circumstance. As the sample is a cellulosic fabric, the appearance of this peak indicated the presence of hydrogen bonding. The presence of free OH at 3739 cm\(^{-1}\) and hydrogen bonded OH stretching at 3415 cm\(^{-1}\) indicates that the cotton fabric had been processed (Benli & Bahtiyari 2015) due to ultrasonication. On peaks at 2953 cm\(^{-1}\) of C-H stretching was also observed (Chung et al. 2004). The peak at 1651 cm\(^{-1}\) was attributed to the stretching vibration of the conjugated and aromatic rings’ C = O groups. C-O-C stretch observed at 1006 between \(\beta\)-glucose unit due to 1,4 glycosidic linkage and pyranose ring within glucose at fingerprint region. There was no other significant differences of FT-IR spectra between conventional and sonicated pretreated cotton fabric.

3.2. Effect on fibre morphologies

The topological modifications of the pretreated and dyed cotton knitted fabric were appraised by SEM and shown in Fig. 2. It was observed that the cotton fiber possesses structural convolutions and ribbon like shape. No noteworthy differences were detected between the traditionally scoured-bleached and sonicated prepared knitted fabric. Additionally, dyed fabric generated by conventional approach and sonication had no significant differences in terms of level of convolution, swelling and also the surface topology as well.

3.3. Water absorbancy and wettability test

The water absorbancy test was performed to evaluate the scouring effect as well as its impact on skin comfort, static build-up, shrinkage, water repellency, and wrinkle healing capabilities. Cotton fabric with a high and constant absorbency are preferred in almost every wet-finishing process (Patnaik et al. 2006). Figure 3 illustrated that when sonicated at 70°C, the ultrasound-aided pretreatment fabric showed the maximum water absorption behavior, 0.30 min, but 0.50, 1.26, and 1.57 min when sonicated at 65°C, 60°C, and 55°C, respectively. Furthermore, the value of 0.5 min for traditionally prepared fabric (at 95°C) was very close to sonication (at 70°C). It is important to note that, despite lowering the temperature by 25°C, ultrasonication results in better absorption than conventional technique.

The wettability of conventional and ultrasonicated fabrics was assessed using wicking performance. Wicking attribute refers to a fabric's capacity to wick moisture away from the body. It determines the wettability of a cloth and how much water it can pass through when in close contact with water. Figure 4 showed the vertical wicking behavior of sonicated knitted cloth. Water wicking was highly rapid initially (less than 1 minute), but it progressively slowed to saturation (Fangueiro et al. 2010). After 30 min, the highest rise in water level was found to be 4.48 inches due to ultrasound effect of cavitation and heating. The majority of ultrasonic energy is wasted by cavitation in low frequency sonication, whereas high frequency sonication wastes energy through heating (Easson et al. 2018). Meanwhile, cavitation may produce “hot spots,” or localized high temperatures, as well as shock waves that produce high pressure capable of shattering chemical bonds (Abou-Okeil et al. 2010).

3.4. Effect of sonication on weight loss

As illustrated in Fig. 5, the weight loss associated with sonicated pretreatment samples is significantly less than that of conventional pretreatment samples. The results of Fig. 5 indicated that the traditional means resulted 7.2% weight loss, but decreased to 6 and 4.9% when the bath chemical was reused twice in a succession. When sonication was performed at 70°C, 65°C, 60°C, and 55°C, weight loss was found 5.7, 5.68, 5.17 and 5.1%, respectively. Sonication resulted in a lower proportion of weight reduction (at 70°C) compared to conventional (at 95°C). The reduction in temperature from 95°C to 70°C achieved through sonication pretreatment offers the possibility of savings in thermal energy.

3.5. Measurement of CIE whiteness

The shade's optimal whiteness is represented by the CIE value, samples treated with sonication techniques had higher whiteness than conventional, however this decreases when the bath chemical was reused, as shown in Fig. 6. The whiteness index for the conventional technique was 72 (at 95°C), whereas the whiteness index for sonication at low temperature (at 70°C) yield 75. Reusing conventional bath chemicals resulted in a decrease in WI (at 95°C, 52). On the other hand, the WI was obtained 63 when the sonication bath chemical was reused at 70°C, which is adequate for dyeing medium to dark shades.

3.6. Effects on fabric strength
The bursting strength of conventionally and ultrasonically pretreated knit fabric samples was illustrated in Fig. 7. The conventional technique's bursting strength was determined to be 198.78 kPa, whereas the sample obtained by multiple reuses of the bath chemical in conventional approaches had 234 kPa and 257 kPa, respectively. The bursting strength of sonicated samples was determined to be 213 kPa at 70°C, 221 kPa at 65°C, 237 kPa at 60°C, and 235 kPa at 55°C, respectively. The bursting strength of the pretreated fabric increased to the same extent as the temperature decreased, as expected by the literature (Sitotaw 2017). Aside from that, the fabric strength values in samples formed by the first and second reuse of bath chemicals are higher than in other samples.

3.7. Color difference for shade matching

Table 3, illustrated that samples dyed at 0.5%, 1.5% and 2.5% shades meet acceptable range of CMC value. It is evident that, sonication technique for dyeing is compatible for shade matching. The CMC values for 0.5, 1.5, and 2.5% shades were 0.89, 0.64, and 0.77, respectively, showing that 1.5 and 2.5% shades passed the normal range, but the 0.5% shade passed the commercial range (Hannan et al. 2019). The picture of the sample dyed with reactive dyes with light shade are given in Fig. 8. According to literature, lower shade percentages provide a greater color difference than higher shade percentages. It is probable that the increased depth of shade was unable to produce noticeable differences in dye absorption (Hannan et al. 2018b). As a result of the sonication procedure, shade matching activity was found to be achievable.

<table>
<thead>
<tr>
<th>Shade%</th>
<th>DL*</th>
<th>Da*</th>
<th>Db*</th>
<th>DE</th>
<th>DC</th>
<th>DH</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5%</td>
<td>0.17</td>
<td>0.67</td>
<td>0.32</td>
<td>0.89</td>
<td>0.52</td>
<td>0.35</td>
</tr>
<tr>
<td>1.5%</td>
<td>-0.59</td>
<td>0.31</td>
<td>0.39</td>
<td>0.77</td>
<td>0.34</td>
<td>0.18</td>
</tr>
<tr>
<td>2.5%</td>
<td>-0.32</td>
<td>0.49</td>
<td>0.28</td>
<td>0.64</td>
<td>0.46</td>
<td>0.27</td>
</tr>
</tbody>
</table>

3.8. Colorfastness properties

As seen in Table 4, ultrasonic dyed samples retained their color better after washing. An excellent or very good to excellent performance was observed when washing fastness of ultrasonic-assisted samples was evaluated. As a result, dyed samples that have been ultrasonically dyed are more washable (Vajnhandl & Le Marechal 2005). Table 4 also revealed that sonication had improved the rubbing fastness in both dry and wet conditions, owing to fabrics smooth surface and absence of hairiness. Colorfastness to perspiration was found to be very good to excellent in sonicated and traditionally dyed samples. Pretreatment of fabric with reused bath chemicals resulted in good and good to excellent color fastness for wash, perspiration, and rubbing. According to Table 4, the color fastness to wash, rubbing, and sweat test results of the treated samples were 4/5 and 5, respectively, even after reusing the bath chemical up to two times. Even though dyed at low temperatures, conventional and reuse of bath chemicals exhibited the same color fastness rating (4) in terms of color fastness to light.

Table 4. Color fastness of ultrasound assisted dyed sample and traditional dyed samples
<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Color Fastness to Wash</th>
<th>Color Fastness to Rubbing</th>
<th>Color Fastness to Perspiration</th>
<th>Color Staining</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Acetate</td>
<td>Cotton</td>
<td>Nylon</td>
<td>Polyester</td>
</tr>
<tr>
<td>Dyed sample (Conventional method)</td>
<td>4</td>
<td>4/5</td>
<td>4/5</td>
<td>5</td>
</tr>
<tr>
<td>Dyed sample (Ultrasound assisted)</td>
<td>4/5</td>
<td>4/5</td>
<td>5</td>
<td>4/5</td>
</tr>
<tr>
<td>Dyed sample (Reuse of bath chemicals)</td>
<td>4/5</td>
<td>4</td>
<td>4/5</td>
<td>4/5</td>
</tr>
</tbody>
</table>

### Conclusion

We have demonstrated that ultrasound process could be beneficial for pretreatment of knit fabric and knit-dyeing process as pretreatment can be carried out at much lower temperature compared to the conventional scouring and bleaching. Additionally, this study examined the reusability of bath chemicals in the pretreatment process. Single bath pretreatment process by sonication improved the whiteness of fabric and reuse of the bath chemical showed satisfactory whiteness index. Sonicated dyeing evolved into three distinct shades: light, medium, and dark. By sonicated dyeing, a pretreatment sample obtained from the reuse of bath chemical meets the medium shade range. The sonicated dyeing did not affect colour fastness properties of knitted fabric. The sonicated method enhances the sustainability of knitted fabric wet processing technique by reducing energy consumption and reusability of bath chemical. As a result, it can be employed as ecofriendly process of pretreatment and dying cotton knitted fabrics with reactive dyes.

### Declarations

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#### Competing interests

The authors have no relevant financial or non-financial interests to disclose.

#### Author contributions

MMUH and SHR designed the research, performed the experiments, analyzed the results, and prepared the draft manuscript. AKM and MMR supervised and assisted the whole research and guided the work and manuscript preparation. MMR and AKM conceived the research, supervised the research design, results interpretation, and manuscript preparation. All authors discussed the results and approved the manuscript.

#### Data availability
The data that support the findings of this study will be available from the corresponding author upon a reasonable request.

**Ethics approval and consent to participate**

The authors are giving ethical approval and consent for the said paper.

**Consent for publication**

The authors are giving their consent for the said paper to be published in ESPR.

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Figures

**Figure 1**

FT-IR spectrum obtained for conventional and sonicated pretreated cotton knit fabric.

**Figure 2**

SEM image of (A) conventionally pretreated knit fabric, (B) and ultrasound assisted pretreated knit fabric, (C) dyed fabric by conventional process and (D) ultrasound assisted dyed fabric.
Figure 3

Effect of absorbency on conventional and sonicated pretreatment cotton knit samples

Figure 4

Effect of ultrasonication time on cotton knit fabric
Figure 5

Weight loss (%) of conventional and sonicated pretreatment samples

Figure 6

Whiteness Index of conventionally pretreated samples along with sonicated samples

Figure 7

Bursting strength of conventional and sonicated samples

Figure 8

Reactive dyed picture of ultrasonic pretreated knitted fabric (A) 0.5% shade, (B) 1.5% shade and (C) with 2.5% shade.